
Publishable Summary for 18SIB10 *chipS*-CALe

Self-calibrating photodiodes for the radiometric linkage to fundamental constants

Overview

Optical power measurement is vitally important for spectrally resolved measurements in photonics across industry, environment, health and science. Using today's costly and work intensive methods based on electrical substitution, the optical power uncertainty is limited by the properties and insufficient stability of current silicon transfer standard detectors. *chipS*-CALe aims to improve and simplify the traceability chain by taking advantage of the intrinsic quantum properties of PQED silicon photodiodes where their improved response to 99.99(9) % is determined by the values of fundamental constants. Developing new and improved technology, accompanied by new metrology, *chipS*-CALe generates for the first time an "NMI-on-a-chip" for optical power. *chipS*-CALe demonstrates that responsivity over a wide spectral range can be predicted based on measurements at one wavelength only.

Need

The European Union had defined Photonics to be one of six Key Enabling Technologies (KET), which is increasingly used in climate monitoring, medical treatment, health and photonic industries, energy saving illumination (LEDs), science, and many more applications. The technological development trend moves in the direction of miniaturisation, more integrated measurement systems and distribution of standalone sensor systems in possibly remote locations. Current metrological systems are not capable of calibrating detectors in integrated systems nor remote locations. Therefore, in both the European technology platform Photonics21's strategic roadmap, "Europe's age of light" from 2021 to 2027 and Quantum Flagship's Strategic Research Agenda March 2020, integration of self-calibrating systems and products are highlighted as one of the technology, research and innovation challenges ahead.

Provided by the best laboratories in the world, the current state-of-the-art spectrally dependent uncertainty of around 0.1 % is limited by the properties and stability of current silicon transfer standard detectors. Previous projects have developed the Predictable Quantum Efficient Detector (PQED), which has proven to have an extremely low external quantum deficiency (EQD) of around 0.01 % and an undetectable drift over 9-10 years. This means that to 99,99 % the responsivity of the PQEDs are determined by the values of fundamental constants. These properties make the PQED a very attractive calibration standard detector and complies well with the low-cost, high-accuracy, transfer standard requested by CIPM's Consultative Committee for Photometry and Radiometry (CCPR). However, the low availability and lack of experimental techniques to independently predict the PQEDs internal quantum deficiency (IQD) has to date prevented it from being exploited as a stand-alone primary standard.

Furthermore, CCPR and EURAMET express the need to measure fundamental constants ratio e/h radiometrically as the ultimate comparison of the two accepted radiometric primary standard detectors, with no preference for either of them, as a contribution to a strengthened and coherent SI system defined by fixed values of fundamental constants.

Objectives

The overall objective of the project is to develop new experimental techniques for optical power measurements over a wide spectral and dynamic range by the production of an "NMI-on-a-chip" detector developed as a self-calibrating silicon photodiode. By "NMI-on-a-chip" we mean a technology suitable for miniaturisation capable of being SI-traceable without external calibration.

The specific objectives are:

1. To develop improved and validated 3D charge-transfer models to predict the PQED internal quantum deficiency. The target prediction uncertainty is 10 % of the internal quantum deficiency value.

2. To develop the best possible PQED photodiodes for cryogenic operation by using the improved 3D models and evaluation of passivation layer materials, passivation strategies and charge increasing techniques. To manufacture a batch of optimised PQED photodiodes and to acquire bare-chip photodiodes for room temperature operation.
3. To develop instrumentation and packaging enabling self-calibration of photodiodes. The photodiodes should be operated in both photocurrent and electrical substitution mode with sufficient sensitivity and equivalence between optical and electrical heating over a temperature range from 20 K to 300 K.
4. To provide traceability of the self-calibrating photodiodes to the revised SI by measuring the fundamental constant ratio e/h to 1 ppm uncertainty at cryogenic temperatures and to 0.05 % uncertainty at room temperature for wavelengths from 400 nm to 850 nm over a dynamic range from 10 nW to 10 mW.
5. To facilitate the take up of the technology and measurement infrastructure developed by the project by engaging standardisation bodies and international organisations (CCPR, CIE, EURAMET and other RMO TC-PR), the measurement supply chain (accredited laboratories, instrument manufacturers) and end users (photonics industry).

Progress beyond the state of the art

Radiometric measurements of radiant power at discrete laser wavelengths are possible with cryogenic radiometers (CR) with an uncertainty down to 0.005 %. Cryogenic radiometers are accurate for most applications, but are bulky, expensive and require a high skill level to operate. Dissemination outside discrete wavelengths is carried out with silicon trap detectors and interpolation functions. The trap detectors' properties and insufficient stability is limiting the comparison agreement at the NMI level to a spectrally dependent dispersion around 0.1 %.

Objective 1: To improve the predicted values of PQED response with 3D simulation software.

The aim has two purposes:

- I. improve the uncertainty in the responsivity to go beyond the IQD losses and
- II. use the model in a new experimental technique to independently extract the photodiode model parameters.

In this way, 3D models are used to put intelligence into the measurement system providing improved understanding and enables new measurement techniques to be developed and applied.

Objective 2: To develop the best possible PQEDs with lower internal losses at cryogenic temperatures.

The IQD in the PQED is determined by simulations to be limited by surface recombination velocity (SRV) and fixed oxide charge (Q_f). The fixed charge is found from standard capacitance voltage (C-V) measurements. The passivation material / process giving the best PQED is effectively found by minority charge carrier lifetime characterisations combined with improved 2D models without going through a full photodiode manufacturing process. The surface characterisation will be performed both at room and cryogenic temperatures.

Objective 3: To develop the required instrumentation and packaging to operate PQED type photodiodes also as a cryogenic electrical substitution radiometer (CESR).

This will realise a self-calibrating dual-mode detector, which can be operated as two independent primary standards with the same absorber. Heat equivalence and signal to noise ratio (SNR) at sufficient accuracy for cryogenic and room temperature operation to meet the target uncertainties in objective 4 will be studied. Developing the PQED also as a CESR will eliminate present calibration limiting errors such as optical window and photon absorption differences from geometry or imperfect blackness. In this dual mode detector, the same number of absorbed photons will generate the signal in either mode of operation.

Objective 4: To demonstrate the metrological applications of the self-calibrating photodiodes at room temperature and cryogenic temperature to unprecedented uncertainties and simplicity.

There are two different routes for self-calibration: i) photocurrent mode with fitted models and ii) dual-mode photodiodes. Exploiting the design of the self-calibrating photodiode, the relative measurement, and PQEDs with improved passivation and prediction models, will allow for radiometric measurement of e/h with 1 ppm uncertainty compared to the 100 ppm uncertainty normally provided by the established standard CESR. The same technology can be used to operate and build self-calibrating photodiodes into applications as an "NMI-

on-a-chip” and by that removing the need to move instruments to the lab for calibration. They can calibrate themselves in their own, possibly remote, and unattended location to a lower cost.

Results

Objective 1: To improve the predicted values of PQED response with 3D simulation software

Simplified 3D simulation models have been developed and fitted to several I-V measurement curves of three different PQED trap detectors (produced in an earlier project) at 476nm, 488 nm and 647 nm using one unique set of parameters for each PQED. Each I-V set consists of up to five different power levels between 100 μ W and 1000 μ W and have been produced by two different laboratories. In the measured change in photocurrent with bias voltage, the IQD vary several orders of magnitude from tens of percent to tens of ppm depending on optical power, bias voltage and beam size. Developing a simplified model was required as more advanced models have computational limitations that would not produce results quickly enough. Still, a remarkably good fit of the predicted IQD to the relative photocurrent measurement at different power levels and bias voltages (in the order of 20 % of the IQD value) was achieved despite the simplified 3D simulation model used. The technique is highly sensitive to beam size and beam energy distribution and the beam shape had to be measured for each wavelength generating the I-V curves. Improvements in a robust beam shape approximation to a gaussian beam in a scalable way was a breakthrough and has given consistent and reproducible fitted results of PQED parameters independent of beam size, wavelength and laboratory performing the measurements. The method with extracted fitted parameters produces values in close agreement with parameters known from the manufacturing using different techniques. The excellent fit shows that the model describes very well the performance of the photodiode and demonstrates that a very good understanding of the physics of the device is achieved. Based on the fitted parameters the spectrally dependent IQD was predicted to range from 100 ppm to below 10 ppm with an uncertainty around 50% of the value. The method has demonstrated its capability of improved sensitivity in the IQD prediction and is ready to be tested on the *chipS*-CALe PQEDs with different parameters. A paper describing the method is submitted to Metrologia.

The work on limitations in PQED prediction uncertainty has revealed that quantum yield (or gain) higher than unity has to be taken into account for wavelengths below 450 nm for high accuracy applications better than 200 ppm. Despite measuring on two PQEDs with different IQD they both produced the same yield value when correcting for the individual differences in IQD. It was demonstrated that the yield can be modelled with an agreement to measurements of around 100 ppm from 400 nm to 450 nm and hence predictability in the PQEDs response is maintained down to 400 nm.

Objective 2: To develop the best possible PQEDs with lower internal losses at cryogenic temperatures

Photodiodes with excellent and record low external quantum deficiency were successfully manufactured in *chipS*-CALe and have thereby reached the most important goal of this project. For the first time PQED photodiodes with SiN_x passivation and a stack of SiO₂ and SiN_x based on previous material research for optimising working PQEDs. Different passivation materials (SiO₂, SiN_x and Al₂O₃) were studied. Surface recombination velocity and fixed oxide charge are, based on simulations, highlighted as the key parameters requiring improvement to optimise working PQED photodiodes. The SiN_x passivation process recipe without any optical absorption and with fixed oxide charge 2-5 times higher than well working qu-candela photodiodes was achieved. Preliminary measurements of their responsivity show that the photodiodes manufactured in *chipS*-CALe are the best for calibration purposes ever produced. High fixed charge improves the linearity of the photodiodes and enables the dual mode techniques to be exploited at higher power and thereby with better signal to noise ratio (SNR). A batch of Al₂O₃ photodiodes is also completed.

A set-up making charge carrier lifetime measurements from 80 K to 300 K has been successfully developed and used to evaluate different process test samples. To the best of the consortium's knowledge, this is the first time lifetime measurements over such large temperature range has been performed. Simulation models were used to separate bulk lifetime and surface recombination velocity from the lifetime measurement. Unfortunately, the lifetime is reduced on all test samples when reducing the temperature. This means that photodiode parameters are changing with temperature and the expected reduction of losses at low temperature will not be fully achieved. From lifetime measurements, prediction of the expected photodiode responsivity, if they were manufactured with the various passivation layers, was done. This demonstrates a new independent measurement technique of the photodiode responsivity from material characterisations and simulations only. At room temperature, the predicted IQD around 1 -10 ppm and 10-100 ppm of the two

produced photodiode types are qualitatively consistent with the first preliminary measurements of photocurrent ratios between previous PQEDs and *chipS-CALe* PQEDs. Validating this requires improvements to the state-of-the-art by an order of magnitude even in relative measurements. However, the improved sensitivity in the method with 3D model fit to I-V measurements may confirm the lifetime method.

The insensitivity of IQD performance with passivation layer thickness enabled us to minimise the reflection losses (for both Al₂O₃ and SiN_x) based on spectroscopic ellipsometry. This resulted in passivation materials with known refractive indices and photodiodes with optimised EQD. The excellent quality of the manufactured photodiodes reduces the need for accurate prediction of the EQD value. Preliminary examinations indicate that responsivity of these photodiodes mounted in a 7 reflection trap structure is VERY CLOSE (1 ppm range) to an ideal photodiode whose responsivity is given by fundamental constants and the applied wavelength only. The work towards improved PQED photodiodes is now published in *Sensors* 2021, 21, 7807 <https://doi.org/10.3390/s21237807>. In the next period work on how good these PQEDs are and comparison between evaluation techniques will be conducted.

Objective 3: To develop the required instrumentation and packaging to operate PQED type photodiodes also as a cryogenic electrical substitution radiometer (CESR).

The purpose of the objective is to develop the technology enabling electrical substitution of optical power measurements on silicon photodiodes. Packaged devices for both room temperature and cryogenic temperatures have been developed based on thermal models with COMSOL Multiphysics. The heating of the photodiode optically is made in the center of the photodiode, whereas electrical heat occurs around the edges. With the current design, simulations show that optical and electrical heat equivalence meeting the target uncertainties is achievable. For room temperature heat equivalence is limited by radiation losses. However, with 14x11 mm² photodiode dimensions target equivalence to 500 ppm was achieved. The heat equivalence between optical and electrical heating is approximately 280 ppm/mm in the positioning of the optical beam. The slope of 280 ppm / mm displacement was confirmed with low noise electrical substitution measurements in WP 4. The sensitivity with beam position depends on the emissivity of the photodiode, which has been carefully measured. An improved packaging with better heat equivalence at room temperature is designed.

At cryogenic temperatures, below 1 ppm heat equivalence in the simulated design has been achieved. Different dual-mode modules have been manufactured according to the design for both room and cryogenic temperature and has been tested for experimental set-ups under objective 4.

Dual mode operation depends on heat equivalence and optimised signal to noise ratio. Evaluation of various temperature sensors for room and cryogenic temperature was conducted to find the best ones for the purpose, and is implemented with the *chipS-CALe* produced photodiodes. New mechanical three-reflection trap detectors optimised for dual mode set-ups have been designed and manufactured. Dual mode detectors based on *chipS-CALe* photodiodes with the best temperature sensors are assembled in the traps and measured.

Objective 4: To demonstrate the metrological applications of the self-calibrating photodiodes at room temperature and cryogenic temperature to unprecedented uncertainties and simplicity

Experimental set-ups for dual-mode photodiode operation are established, one for room and one for cryogenic temperatures. The work has triggered new ideas and two different types of measurement procedures have been developed and tested, compared to the initially planned one. Furthermore, three different calculation algorithms are tested. Time constants in the dual mode modules have been measured both optically and electrically. There is a good agreement between the modelled and measured time constants even though the deviation was a bit higher than expected. The high fixed charge of *chipS-CALe* photodiodes ensures linearity at higher power levels and the room temperature dual mode experiment can therefore be run with improved SNR. Room temperature dual mode experiments have improved significantly in terms of signal-to-noise ratio. Work on thermal stabilisation, electrical shielding and improved measurement bridge have resulted in excellent measurement of the IQD of the assembled three-reflection trap dual-mode *chipS-CALe* photodiode with a Type A uncertainty of 13 ppm for a power level of 1250 μW in a 6 hour long measurement sequence with the calculation algorithm producing the lowest uncertainty. The excellent SNR enabled the experimental verification of the expected non-equivalence resulting in an IQD calculation changing 280 ppm/mm with beam position. The work further revealed that dust particle on the optical window influenced the IQD measurement with 130 ppm and that design care has to be taken into account when measuring in dual mode on detectors in a trap structure. The residual optical heating of the second photodiode in the trap structure must be compensated in order not to influence the heating profile and read out of the target photodiode.

The improved resolution and uncertainty in the measurement show a discrepancy between the two measurement methods up to 700 ppm at 500 μ W that previously was hidden in the noise of the experiments. The demonstration of the new measurement procedure paves the way for a possibly third measurement procedure by implementing the dual mode detector in a closed feedback loop configuration. The measured IQD is higher than the expected IQD from the manufacturing of the photodiodes. Three different calculation algorithms agree within their combined uncertainties. Optimisation and exploration of achievable uncertainty of the method will be the main focus ahead. The room temperature dual mode work is submitted for publication in Metrologia.

Measurements of dual mode detectors at cryogenic temperatures will have improved heat equivalence to better than 1 ppm and has reduced thermal noise, lower heat capacity, increased heat conductivity, lower time constant and better photodiode linearity as compared to room temperature. Initial measurements of dual mode trap modules with *chipS·CALe* photodiodes are done at different cryogenic temperatures between 35 K and 60 K. The measurements have revealed that improved thermal stabilisation is needed at the cryogenic measurements and heat cross talk between photodiodes in the trap detector has to be reduced. Work on stabilising the temperature of the dual mode detector is progressing. There is a difference in noise level between the two methods for the dual mode experiment. Typical Type A uncertainty of 50 ppm is achieved with the open circuit method and 6 ppm in forward bias mode.

Impact

Many consortium members demonstrated project results in International Conference on New Developments and Applications in Optical Radiometry (NEWRAD 2020, Boulder, CO, US) which was postponed as a digital conference in 2021. The project coordinator was invited by the scientific organising committee to give an oral presentation. In total 7 contributions from chipSCALE project were presented at the virtual conference.

The project website was developed at the beginning of the project to share our latest publishable results (<http://chipscale.aalto.fi/>), such as the publishable summary, publications in journals, posters, presentations from conferences, etc., and the project's page on the ZENODO repository (https://zenodo.org/communities/empir_chipscale/?page=1&size=20) allows the publishing of documents with a unique DOI number.

The material research in *chipS·CALe* has helped SINTEF, as a small-scale detector manufacturer, optimise the manufacturing processes and has resulted in a new SiNx production recipe. Two new and different fabrication processes of PQED type photodiodes are established and SINTEF is now able to offer this technology at full manufacture level. The successful manufacturing of the Mise-en Pratique approved primary standard PQEDs closes the availability gap of these devices and opens up for wider exploitation of the PQEDs. The work towards manufacturing improved PQEDs with a production batch predicted responsivity is published in Sensors. The working principle of the PQED photodiode is explained in an animation video produced in the project.

Impact on industrial and other user communities

The production volume of the European Photonics industry accounted for € 69.2 billion in 2015. Industry in general requires accurate and cost-efficient calibration methods to maintain traceability to an SI unit of their methods and equipment. Working out an exploitation plan and making the self-calibrating photodiodes commercially available are the most important criteria for uptake of the technology developed in *chipS·CALe*. Commercialisation of developed products is likely to be targeted in a follow up Support for Impact Project (SIP) project. The planned animation video will help users understand how the devices work and make it appealing to use the devices. This will also be instrumental in the promotion of the project in scientific talks, on the website and in the approach to the wider user community.

Photonics21 is one of the European Technology Platforms, supporting the Key Enabling Technologies (KET) defined by the EU, and has more than 2500 members from the photonic industry, research institutes, academia and public service. The established contact between the consortium and Photonics21 simplifies the transfer of knowledge about project outputs to this important technology platform. In Photonics21 WG5 strategic roadmap "Europe's Age of Light! How photonics will power growth and innovation" for the period 2021-2027 they have already implemented the ideas of *chipS·CALe* and request "maintenance-free, self-calibrating sensors" as both a technology challenge and a research and innovation challenge for optimised value.

Impact on the metrology and scientific communities

The principles and methods developed in *chipS*-CALe will support and strengthen the implementation of the new SI system and the radiometric community's position within the SI. The CCPR has requested the possibility of making radiometric measurement of fundamental constants as will be demonstrated here with unprecedented accuracy by comparing two independent and inherently different primary standards in one device. The CCPR is also requesting better and more stable measurement standards. CCPR Intercomparison in the 300 nm to 1000 nm spectral range is postponed because present photodiodes are not sufficiently stable. Previous PQEDs have shown an undetectable drift over 10 years. With the commercialisation of the *chipS*-CALe photodiodes the community will have a new chip-scaled device for measuring optical power with unprecedented accuracy in possibly remote operation.

The approved Researcher Mobility Grant from a researcher from PMOD/WRC is a demonstrator of an early uptake of *chipS*-CALe technology by the scientific community. PMOD/WRC plays a crucial role in maintaining quality standards in global climate monitoring programmes. The purpose of the RMG is to improve the standard laboratory measurements from the existing 2 % to better than 0.5 %.

The validated 3D simulation tool will be used to establish a new service for owners of PQEDs based on customer's own relative characterisations. The new service would support laboratories to make independent realisations based on PQEDs alone. The self-calibrating device will also help a number of project partners to reduce their CMC uncertainty for in-house customer calibration service. In a perspective beyond *chipS*-CALe, other European NMIs can develop their capacity based on the self-calibration technology. Prediction of PQED response can be made from characterisations done at one wavelength with an extended characterisation interval due to the excellent stability.

There exist concrete plans in granted projects to exploit PQED photodiodes in conjunction with photonic integrated circuits (PICs) as an absolute embedded standard for extended dynamic range of optical power. This is intended as a first demonstrator and one possible way of exploiting photodiodes in integrated photonics which existing standards are incapable of doing. The embedded reference may be exploited in the future as this technology becomes more mature and wide-spread in various applications.

The project coordinator informed the EURAMET TC-PR about the project achievements in a workshop in conjunction to the annual 2021 TC-PR meeting including emphasis on the successful manufacturing of improved predictable quantum efficient detectors. A more extensive presentation of the results is scheduled for the EURAMET TC-PR in January 2022.

Impact on relevant standards

CIE is the international standardisation committee for light and lighting. CIE – Division 2: Physical Measurement of Light and Radiation has requested that the new experimental techniques developed in *chipS*-CALe are fed into the ongoing revision of TC2-81 Update of CIE065:1985 (Absolute Radiometers). CIE welcomes the idea of an "NMI-on-a-chip" and has agreed to join the stakeholder committee to closely follow the progress of *chipS*-CALe. The technical committee leader of TC2-81, a consortium partner in *chipS*-CALe, presented the project at the CIE 29th Quadrennial Session in Washington June 2019, and will ensure that project outputs can be effectively implemented in the new standards. PQEDs could be exploited in future standards and measurement equipment of interest to CIE. The consortium has given input to the ongoing revision of CIE TC-81 CIE065:1985 Absolute radiometers. Implementation in this global standard is expected to further promote and trigger the use of these devices.

Longer-term economic, social and environmental impacts

Photonic sensors are key enablers in a wide range of industrial manufacturing and service sectors including: healthcare, surveillance, and automotives. The resultant leverage makes photonic metrology and sensors a multi-billion euro industry. Improved sensors and simplified traceability will contribute to an improved efficiency in the photonic sensor industry.

The fraction of the population above 65 years age is increasing and this will put more pressure on the health care system. Optical methods are used increasingly both for diagnosis and therapy. Supporting optical methods with improved and simplified calibrations will contribute both on the improved diagnosis and optically based therapy side, as they are known to be faster and less invasive compared to previous surgical methods. About 2/3 of the Essential Climate Variables (ECV) used to monitor impact of climate change require some sort of optical measurement. SI traceable measurements over decadal timescales require new instrumentation to detect small trends in ECVs from a background of natural variability. A self-calibrating instrument making climate quality measurements in the field, ideally in an autonomous manner, would meet this requirement and

is mentioned as an important application of basic science in the environmental roadmap for photometry and radiometry. The self-calibration detector proposed in this project is a first step towards such an instrument, and will have a major impact on the quality of Earth Observation data.

List of publications

[1] Koybasi O. et al. High Performance Predictable Quantum Efficient Detector Based on Induced-Junction photodiodes Passivated with SiO₂/SiN_x. *Sensors* **2021**, 21, 7807. <https://doi.org/10.3390/s21237807>

This list is also available here: <https://www.euramet.org/repository/research-publications-repository-link/>

Project start date and duration:		01 June 2019 (36 months + 6 months extension = 42 months)
Coordinator: Dr Jarle Gran, JV		Tel: +47 64 84 84 45
Project website address: http://chipscale.aalto.fi/index.html		E-mail: jag@justervesenet.no
Internal Funded Partners:	External Funded Partners:	Unfunded Partners:
<ul style="list-style-type: none"> 1. JV, Norway 2. Aalto, Finland 3. CMI, Czech Republic 4. CNAM, France 5. INRiM, Italy 6. Metroserf, Estonia 7. PTB, Germany 8. TUBITAK, Turkey 	<ul style="list-style-type: none"> 9. IFE, Norway 10. SINTEF, Norway 11. USN, Norway 	
18SIB10-RMG1 chipS·CALe: PMOD/WRC, Switzerland		